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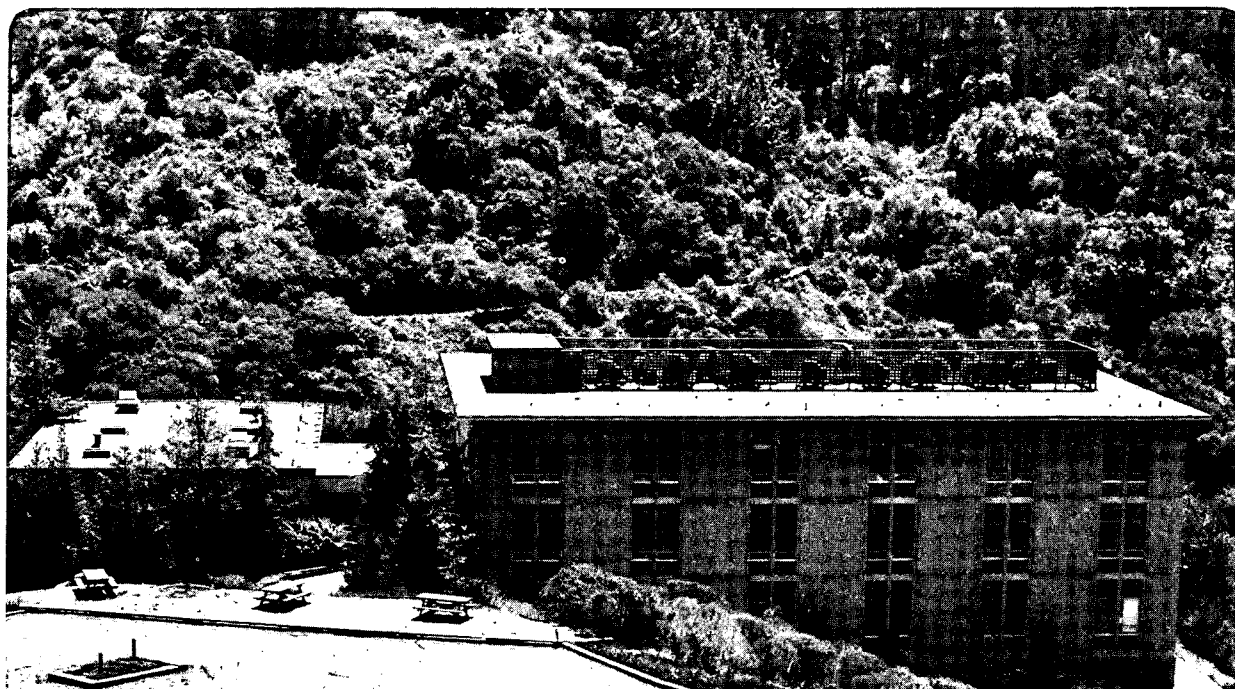
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J.G. Ulan, C. Schooley, and R. Gronsky

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**MODIFIED EMBEDMENT PROCEDURE FOR MICROTOMY
OF LARGE PARTICLE ZEOLITES**

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MODIFIED EMBEDMENT PROCEDURE FOR MICROTOMY OF LARGE PARTICLE ZEOLITES

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Abstract

A method is described to strengthen the binding of organic resin to inorganic zeolite, allowing large particles to be microtomed. For FeZSM-5 aggregates the particle size limit increased from 3 μm to greater than 20 μm in diameter by application of this method. This technique can be applied to a variety of oxide powder samples, extending the utility of microtomy as a materials science tool.

Introduction

In one form of microtomy, particulate samples are first embedded in resins and then electron transparent thin sections are sliced. The entire particle can then be examined by TEM for compositional and structural information. However, there is a limit to the size of particles that can be microtomed since larger particles tend to dislodge from the embedding resin when sectioned. In our particular case the effect of preparing Fe-zeolites in the presence of various alkali cations was under study. These zeolites grow as aggregates of crystallites which have a size range from 2 μm to 10 μm and several samples had extensively fused aggregates, resulting in a total particle size that was often much greater than 100 μm . A previously successful procedure for preparing microtomed zeolite samples for TEM (Csencsits et al., 1985) failed for all but the smallest aggregates. We developed a new procedure which employed a coupling reagent during embedment. The interface between zeolite aggregates and resin is strengthened allowing it to withstand the force of cutting during microtomy. The use of the coupling reagent DOW Z-6040 to prevent pullout during microtomy was reported by Swab and Klinger (1988) for TEM specimen preparation of optical multilayer devices. We are reporting its use for particulate samples.

Materials and methods

ACRYLIC EMBEDDING FOR INTERMEDIATE SIZED PARTICLES

The samples were crushed with a mortar and pestle. A few mg of crushed iron zeolite were placed in the tip of a 00 gelatin capsule (LR White penetrates most polyethylene molds); the capsule was filled with the acrylic resin LR White (hard grade) and left for 24 hours at room temperature under house vacuum. The blocks were heat cured at 94 -100 $^{\circ}\text{C}$ for 1.5 to 2 hrs. After cooling to room temperature the gelatin capsules were removed by soaking in water. The block faces were microtomed as described elsewhere (Csencsits et. al. 1985).

COUPLING REAGENT/EPOXY EMBEDMENT FOR LARGE PARTICLES

Samples were crushed with a liquid nitrogen cooled mortar and pestle as cooling the material increased its brittleness and produced smaller fragments. A 0.3 wt. % (g/g) aqueous solution of 3-glycidoxy-propyltrimethylsilane (DOW Z-6040) was prepared, the pH adjusted to 3-4 with acetic acid and stirred for 45 minutes. A 120 mg sample of crushed iron zeolite was added to 30 ml of the 0.3 wt % solution of Z-6040 and again stirred for 45 minutes. The mixture was filtered and the solid iron zeolite dried at 100 $^{\circ}\text{C}$ for 4 hrs.

The resin components were mixed in the following proportions and order; 13.5 ml of Araldite 6005, 11.5 ml of dodecenylsauccinnic anhydride (DDSA) and 0.5 mL of benzyldimethylamine (BDMA). The resin mixture can be stored up to four days in a refrigerator. To avoid water condensation the container was tightly capped and brought to room temperature before uncapping. (Resin components are hazardous and should be handled with appropriate safety precautions). Air bubbles, which can form during mixing, prevent polymerization so the mixture was degassed using a house vacuum. The epoxy resin, Araldite 6005 was chosen because it is not as sensitive to moisture as other epoxy resins, (e.g., Spurr) making it easier to use. The proportions of resin components were modified from standard proportions reported in the literature (Glauert, 1974). BDMA was used as an accelerator to improve the viscosity of the resin and achieve better polymerization (Glauert, 1987). An epoxy resin must be used with the zeolites after they are treated with Z-6040 Attempts to embed the treated zeolite in LR White failed

A 20 mg sample of the iron zeolite was dehydrated with two washes of 100% methanol (3 ml, for 10 minutes each) and then two washes of propylene oxide (3ml, for 10 minutes each) and stored under propylene oxide to avoid rehydration. The iron zeolite was infiltrated with a 1 part propylene oxide (3.5 ml) and 1 part resin mixture (3.5ml) for 24 hours at room temperature. Then the propylene oxide/resin mixture was pipetted off and the iron zeolite was infiltrated with 100% Araldite resin mixture for 24 hours at room temperature. The Araldite 6005 resin mixture was pipetted off and small amounts of iron zeolite were transferred to the tips of an embedding molds after which the molds were filled with resin mixture. The molds were degassed under house vacuum before curing at 60 °C for 24- 48 hours. The blocks were microtomed as described previously.

Results and discussion

By increasing the LR White infiltration time it was possible to section aggregates up to 5 μm in diameter. We would suggest trying this simple modification first because the procedure is still relatively fast and LR White acrylic is easy to handle and less hazardous than the multicomponent, viscous epoxy resin.

By using a coupling reagent and Aradite 6005 it was possible to section aggregates larger than 20 μm in diameter. Figures 1 shows samples of aggregates of FeZSM-5 treated with K^+ embedded using the Z-6040/Araldite 6005 procedure. The morphology and crystallinity of the center and surface of the aggregates can be compared, and the composition at various places in an aggregate can be measured. This information is unattainable with crushed and dispersed samples because the centers of the aggregates are too thick to study. The use of a coupling reagent to extend the size range of particles that can be sectioned should be useful for other oxide powder samples, extending the utility of microtomy as a materials science tool.

Acknowledgements

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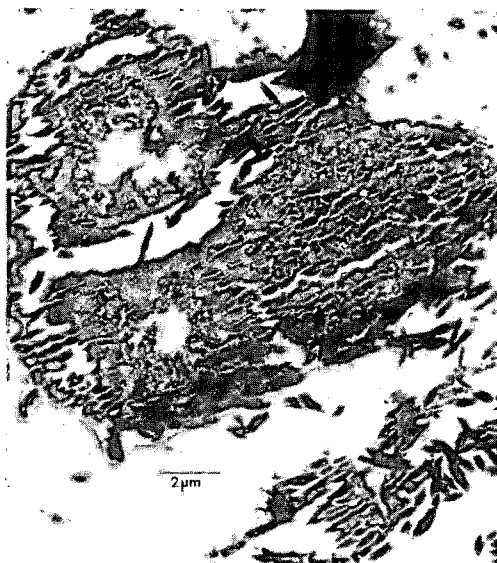


Figure 1: TEM image of large FeZSM-5 aggregates prepared using Z-6040/Araldite 6005 method.

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